



In re application of
Mika Perälä et al.

Serial No. 10/019,962
Filed July 4, 2000

Art Unit: 1712
Examiner: Jeffrey B. Robertsson

COMPOSITION TO BE USED IN PAINTS

DECLARATION

I, Mika Perälä, Master of Science in Chemical Engineering, being employed from February 23, 1998 as a R&D Chemist in NOR-MAALI OY in Finland and being one of the inventors of the invention described and claimed in the subject patent application, depose and state the following facts with respect to the non-obviousness of the composition to be used in paints of the present invention as regards.

Comparative tests were carried out using paint formulation based on the patent of Gasmena with and without silicon containing polyether component (Kaneka) and the paint formulation made in accordance with present invention.

Paint formulation by Gasmena

A first part of a heat ablative coating composition was prepared by combining 2 grams(g) of the epoxy functional silane compound (Silquest A-187), 7,6 grams of xylene, 15 g of the epoxy resin (Erisys RF-50), 3,4 g of the siloxane (Dow Corning 3074), 0,6 g of the organotin compound (DBTL), and 0,3 g of the organozinc compound and mixing the ingredients together until uniform. To such first part ingredients were added 12 g clay filler, 0,8 g ethanol, 2,7 g glycol ether acetate, 13,3 g titanium dioxide pigment, 2,3 g black oxide pigment, 12 g talc filler, 18 g ceramic fiber filler, 0,9 g deionized water, 8,4 g xylene and 0,3 g thixotropic agent. The first part ingredients were mixed together at room temperature until uniform.

A second part of the heat ablative coating composition was prepared by combining 66 g silicon containing polyether (Kaneka), 0,6 g thixotropic agent, 5 g ceramic fiber filler, 16 g clay filler and mixing the ingredients together until uniform. To such second part ingredients were added 7,6 g xylene, 2,4 g aminosilane (Silquest A-1100); and 2,4 g amine catalyst (Ancamine K-54). The second part ingredients were mixed together at room temperature until uniform.

A heat ablative coating composition was prepared by combining the first and second part ingredients together and mixing the combined ingredients at room temperature until uniform.

Paint formulation by Gasmena without silicon containing polyether

A first part of a heat ablative coating composition was prepared similar way as described above.

A second part of a heat ablative coating composition was also prepared similar way but without silicon containing polyether (Kaneka). Amount of xylene was increased in the formulation to get the viscosity into the right level for application. Additional xylene was approximately 14,6 g.

A heat ablative coating composition was prepared by combining the first and second part ingredients together in stoichiometric ratio like in the example above and mixing the combined ingredients at room temperature until uniform.

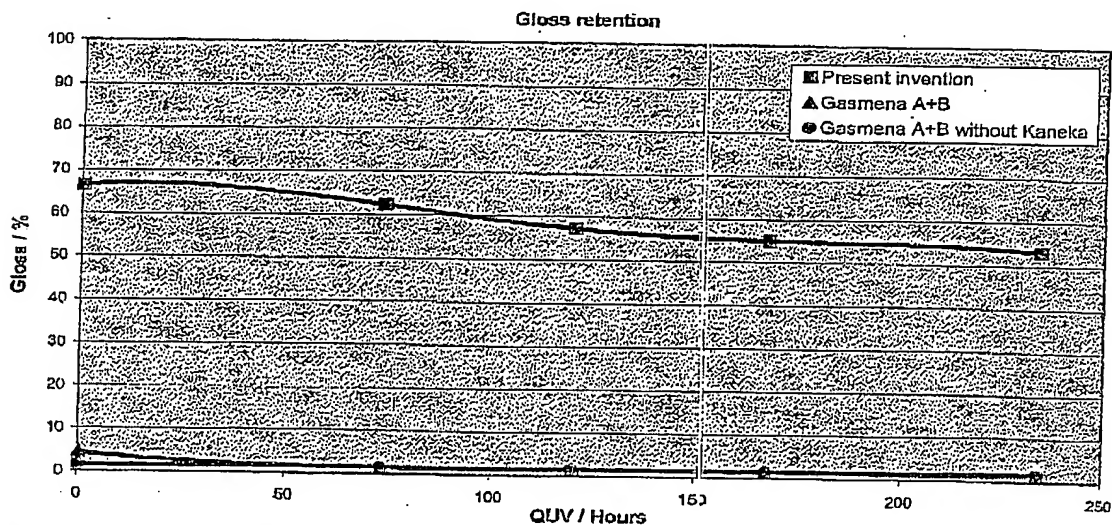
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Paint formulation by present invention

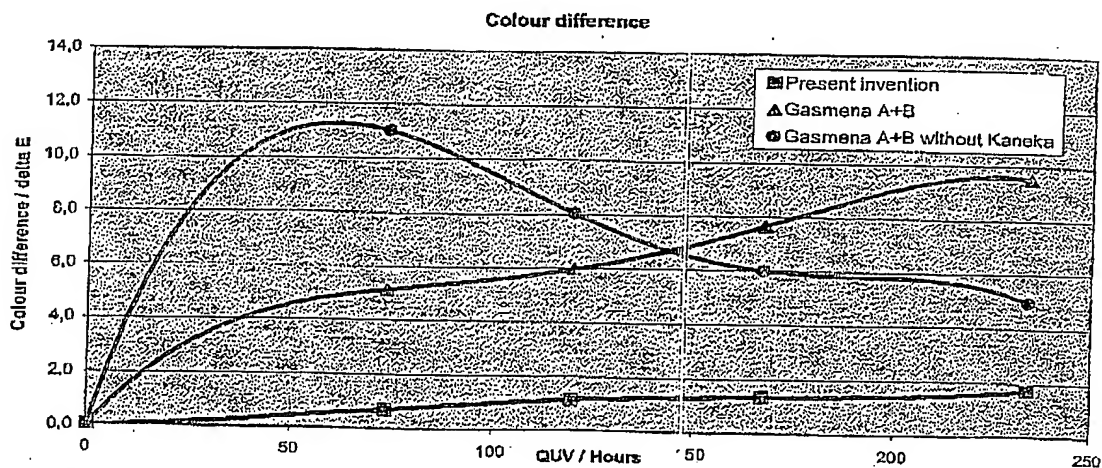
The polysiloxane paint was made in accordance with example 3 by present invention.

UV-resistance

Weathering test for the coatings by Gasmene and present invention was made with the QUV accelerated weathering tester according to standard ASTM G-53 (UVA 340 nm). Gloss retention and colour difference results are shown in the pictures 1 and 2.



Picture 1. Gloss retention of coating formulations by Gasmene and present invention.



Picture 2. Colour difference of coating formulations by Gasmene and present invention.

According to the results from weathering tests it can be seen that by taking out the silicon containing polyether from formulation there are still significant differences in the performance of the coating systems in question.

Composition differences of the paint formulations

Composition differences of the paint formulations by Gasmena and present invention are shown in the table I. Gasmena figures are taken from the patent and the figures of the present invention are calculated from example 3 with stoichiometric mixing ratio. The figures for Gasmena without silicon containing polyether are obtained by replacing polyether component with additional amount of xylene and then calculated from stoichiometric mixture.

Table I: Composition differences of the paint formulations by Gasmena and present invention.

	Epoxy functional silane	Siloxane	Epoxy	Polyether (Kaneka)
Gasmena (range)	0,5-5 %	0,5-5 %	1-20 %	20-45 %
Gasmena (preferred)	1 %	2 %	8 %	35 %
Gasmena (without Kaneka)	1,4 %	2,5 %	10,9 %	---
Present invention	4,4 %	27 %	23 %	---

Based on the results from table I it can be seen that ingredients similar to present invention (epoxy functional silane and siloxane) are at the level of additives in composition not part of a binder system.

In the present invention ingredients are non-aromatic epoxy resin, polysiloxane, epoxy functional silane and the hardener. By using the ingredients mentioned above as a binder system it is possible to manufacture and apply the paint without adding silicor. containing polyether. By this way it is possible to obtain high solids content paint with a very low VOC-level. The use of silicon containing polyether is not reasonable based on the weathering tests. Silicon containing polyether causes a rubber-like surface in the paint film which is not desirable due to its inability to tolerate scratches and mechanical stress.

The curing properties of the paint compositions were determined with the ball-type linear drying time recorder. The determination was made with non-pigmented laquers of Gasmena without silicon containing polyether and present invention to find out the actual curing rate of the binder systems. Results from the test are shown in the table II.

Table II: Drying times for the laquer versions of Gasmena without silicon containing polyether and present invention.

	Surface dry	Through dry	Hard dry
Gasmena (without Kaneka)	1,5 hours	3,5 hours	> 24 hours
Present invention	30 minutes	1,5 hours	4 hours

Even without silicon containing polyether the curing speed of Gasmena composition is considerably slower compared to the present invention.

The patents of Eklund and Iwamura represents different kind of technology one being powder coating application and other being stoving paint resin composition. Although similar type of resins mentioned and at some special cases used in those applications we do not see any relevance to the matter because of the totally different technologies.

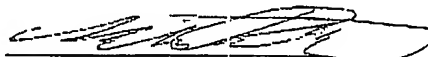
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Conclusion

In the present invention ingredients are non-aromatic epoxy resin, polysiloxane, epoxy functional silane and the hardener. The coating composition according to the patent of Gasmena is based on the use of silicon containing polyether and resorcinol modified epoxy resin. The ingredients of the present invention are used for different purposes than the Gasmena composition. As stated in the Gasmena patent, epoxy functional silane acts as an adhesion promoter and siloxane is added to provide heat and fire resistance. While in the present invention polysiloxane and non-aromatic epoxy resin forms a binder system where the epoxy silane works as a crosslinker and siloxane compound is for better weatherability. Also when comparing the amounts of ingredients used in Gasmena, epoxy functional silane and siloxane compounds are at the level of additives and do not reflect levels that would be used in a binder system as in the present invention. Based on the results from the weathering test and drying time test one can see that leaving the polyether component out of the formulation does not mean that the paint composition by Gasmena renders obvious the present invention. Similar epoxy resins mentioned by Eklund and Iwamura are not relevant because of the different technologies used in the applications.

I further hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, both, under §1001 of Title 18 of the United States Code and that such wilful statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 21.01.2005


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Comparative tests were carried out using binder system based on the patent of Kuriyama et al (Example 10) and the binder system made in accordance with present invention.

Binder by Kuriyama et al (Example 10)

To bisphenol A type epoxy resin (Epikote 828, 100 parts by weight) is added dibutyl tin dilaurate (0,5 part by weight), and to the mixture are added liquid hydroxyl-terminated, linear polydimethylsiloxane (Silopren C 1, 30 parts by weight) and 3-glycidoxypropyltrimethoxysilane (Silquest A-187, 1 part by weight) with stirring. The mixture is heated (+80°C) for 6 hours. The epoxy resin thus obtained is a milky white liquid. The composition has a viscosity of 32 000 cP.

Binder by present invention

To non-aromatic epoxy resin (Epodil 757, 100 parts by weight) is added liquid methoxy-functional polysiloxane (Dow Corning 3074, 116,5 parts by weight) and 3-glycidoxypropyltrimethoxysilane (Silquest A-187, 20,6 parts by weight) with stirring. The binder thus obtained is clear liquid. The composition has a viscosity of 64 cP.

Curing

Both binders obtained with procedures mentioned before were cured with a modified aliphatic polyamine mentioned in patent by Kuriyama (Ancamine 1784) and the hardener described in example 3 in present invention.

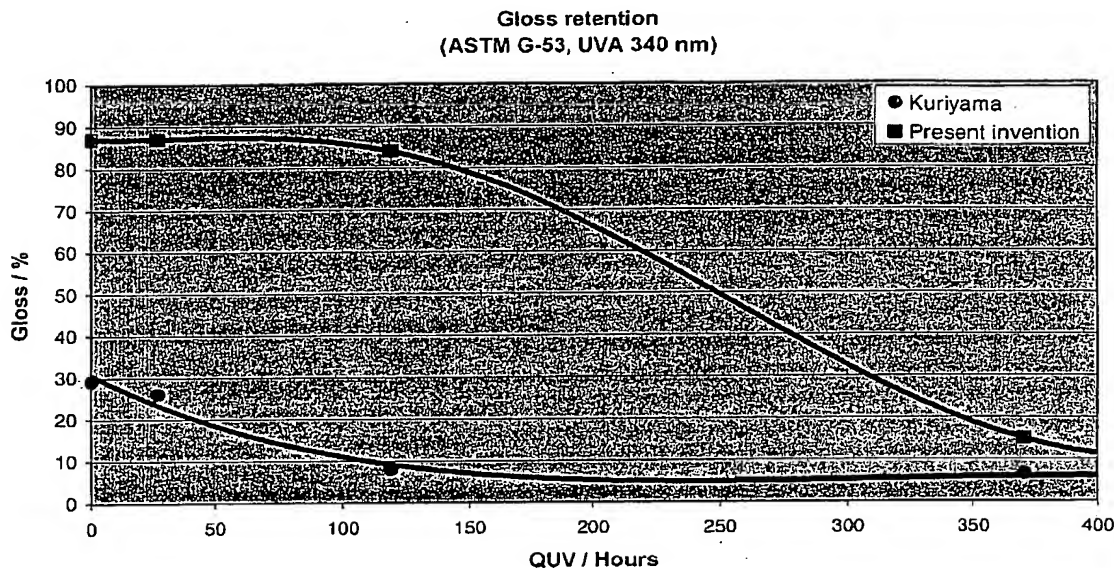
Curing of the epoxy resin obtained by Kuriyama was very slow with modified aliphatic polyamine. After 96 hours curing in 23°C and 48 hours stoving in 50°C the paint film was still sticky. Also the surface of the coating was greasy. After mixing with the hardener by present invention a lot of agglomeration occurred and it was impossible to apply the coating.

The binder obtained by present invention was cured with modified aliphatic polyamine with same procedure. After 96 hours curing in 23°C and 48 hours stoving in 50°C the paint film was cured and not sticky like the binder by Kuriyama. Combination with the hardener by present invention gave cured paint film after 72 hours.

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UV-resistance

Weathering test for the coatings in question was made with the QUV accelerated weathering tester according to standard ASTM G-53 (UVA 340 nm). The gloss retention of the binders cured with a modified aliphatic polyamine is shown in the picture 1. The UV-resistance of the paint films obtained with the hardener described in example 3 in present invention was not measured because the binder by Kuriyama did not combine with it.



Picture 1. Gloss retention of binders cured with modified aliphatic polyamine.

Conclusion

Composition to be used in paints made by present invention differs from the resin obtained by Kuriyama with the procedure how the resin part is made. In other words reaction with elevated temperature and catalyst is not needed. In present invention the silanol functionality is not necessary in organopolysiloxane. Methoxy-functional polysiloxane used in present invention can not be comprised of the formula of an organopolysiloxane by Kuriyama. Use of organosilane is limited to epoxysilane in present invention. In the patent of Kuriyama there are no limitations for the organosilane compound. Also the viscosities of the compositions are in totally different range. If a pigmented paint is made from resin by Kuriyama it has to be thinned with solvents. The paint made by present invention can be manufactured and also applied by spraying without adding any solvents.

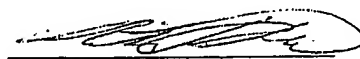
Curing properties of the resin by Kuriyama differs considerably from the binder by present invention with the hardener mentioned by Kuriyama (modified aliphatic polyamine). In practice binder with such curing properties is not useful for paint purposes.

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UV-resistance is better with the composition by present invention according to the weathering test. This is one of the main issues of the present invention of the composition to be used in paints.

I further hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, both, under §1001 of Title 18 of the United States Code and that such wilful statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 03.03.2004



Mika Perälä

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